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Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE

UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No. 2000 1545 | *Total Pages*

First Named Inventor or Application Identifier

Giovanni Pietro CHIAVAROTTI et al.

Express Mail Label No.:

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, D.C. 20231

1. [X] Fee Transmittal Form
(Submit an original, and a duplicate for fee processing)

2. [X] Specification *[Total Pages -10]*
(preferred arrangement set forth below)
- Descriptive title of the Invention
- Cross References to Related Applications
- Statement Regarding Fed sponsored R & D
- Reference to Microfiche Appendix
- Background of the Invention
- Brief Summary of the Invention
- Brief Description of the Drawings *(if filed)*
- Detailed Description
- Claim(s)
- Abstract of the Disclosure

3. [] Drawing(s) *(35 USC 113)* *[Total sheets -]*

4. [X] Oath or Declaration *[Total Pages - 3]*
a.1. [] Newly executed (original or copy)
a.2. [] Unexecuted
b. [X] Copy from a prior application (37 CFR 1.63(d))
(for continuation/divisional with Box 17 completed)
[Note Box 5 below]
i. [] DELETION OF INVENTOR(S)
Signed statement attached deleting inventor(s) named in the prior application, see 37 CFR 1.63(d)(2) and 1.33(b).

5. [X] Incorporation By Reference
(usable if Box 4b is checked)
The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under Box 4b, is considered as being part of the disclosure of the accompanying application and is hereby incorporated by reference therein.

6. Microfiche Computer Program *(Appendix)*

7. []Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
a. [] Computer Readable Copy
b. [] Paper Copy (identical to computer copy)
c. [] Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

8. [] Assignment Papers (cover sheet & document(s))
9. [] 37 CFR 3.73(b) Statement [] Power of Attorney
(when there is an assignee)
10. [] English Translation Document *(if applicable)*
11. [X] Information Disclosure Statement (IDS)/PTO-1449
[] Copies of IDS Citations
12. [X] Preliminary Amendment
13. [X] Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
14. [] Small Entity Statement(s)
[X] Statement filed in prior application, Status still proper and desired
15. [] Certified Copy of Priority Document(s)
(if foreign priority is claimed)
16. [X] Other: Claim of Priority

17. If a CONTINUING APPLICATION, *check appropriate box and supply the requisite information:*
[] Continuation [X] Divisional [] Continuation-in-part (CIP) of prior Application No. 09/357,300

18. CORRESPONDENCE ADDRESS

WENDEROTH, LIND & PONACK, L.L.P.
2033 "K" Street, N.W.
Suite 800
Washington, D.C. 20006
Phone:(202) 721-8200
Fax:(202) 721-8250

THE COMMISSIONER IS AUTHORIZED TO CHARGE ANY DEFICIENCY IN THE FEE FOR THIS PAPER TO DEPOSIT ACCOUNT NO. 23-0975.

November 8, 2000

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of	:	
Giovanni Pietro CHIAVAROTTI et al.	:	
Serial No. NEW	:	**Attn: Application Branch**
Filed November 8, 2000	:	**Attorney Docket No. 2000_1545**
PROCESS FOR PRODUCING AN ELECTRODE AN USE OF THE ELECTRODE **(Rule 1.53(b) Divisional of Serial No. 09/357,300, Filed July 20, 1999)**	:	

PATENT OFFICE FEE TRANSMITTAL FORM

Assistant Commissioner for Patents
Washington, DC 20231

Sir:

Attached hereto is a check in the amount of $355.00 to cover Patent Office fees relating to filing the following attached papers:

Rule 53(b) application $355.00

A duplicate copy of this paper is being submitted for use in the Accounting Division, Office of Finance.

The Commissioner is authorized to charge any deficiency or to credit any overpayment associated with this communication to Deposit Account No. 23-0975, with the EXCEPTION of deficiencies in fees for multiple dependent claims in new applications.

Respectfully submitted,

Giovanni Pietro CHIAVAROTTI et al.

By _____
Michael R. Davis
Registration No. 25,134
Attorney for Applicants

MRD/sls
WENDEROTH, LIND & PONACK, L.L.P.
2033 K St., N.W., Suite 800
Washington, D.C. 20006
Telephone (202) 721-8200
November 8, 2000

[Check No. 40676]

2000_1545

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PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, DC 20231

Sir:

Please amend the above-identified divisional application as follows:

IN THE SPECIFICATION:

Page 1, after the title of the invention, please insert

--CROSS-REFERENCE TO RELATED APPLICATION

The present application is a division of Serial No. 09/357,300 filed July 20, 1999.--

IN THE CLAIMS:

Cancel, without prejudice to the subject matter involved, claims 1-14 and 21-31.

REMARKS

The present divisional application is being filed as a result of a restriction requirement in the parent application, Serial No. 09/357,300 filed July 20, 1999. The divisional application is directed to claims 15-20, which were not elected in the parent application.

Respectfully submitted,

Giovanni Pietro CHIAVAROTTI et al.

By _____
Michael R. Davis
Registration No. 25,134
Attorney for Applicants

MRD/sls
Washington, D.C.
Telephone (202) 721-8200
Facsimile (202) 721-8250
November 8, 2000

THE COMMISSIONER IS AUTHORIZED TO CHARGE ANY DEFICIENCY IN THE FEE FOR THIS PAPER TO DEPOSIT ACCOUNT NO. 23-0975.

PROCESS FOR PRODUCING AN ELECTRODE AND USE OF THE ELECTRODE

BACKGROUND OF THE INVENTION

The present invention relates to a process for producing an impermeable or substantially impermeable electrode, for example, electrolytic capacitors, supercapacitors or batteries.

It is known in the field of electrolytic capacitors to use metal foils as cathodes. Therein the metal foils are etched chemically or electrochemically. The etching penetrates into the material body of the metal foil whereby the surface and, thus, the specific capacitance, of the foil is increased. Due to the etching, the electric resistance, for example of an aluminum foil, in comparison to a non-etched foil, is increased by a factor which is a function of the quantity of the metal removed during the etching process. In addition, the metal foil loses mechanical strength. The natural oxidation which takes place with this treatment yields a capacitance which is a function of the dielectric constant of the metal itself and the treatment performed on the metal foil.

SUMMARY OF THE INVENTION

In contrast, according to the present invention the desired capacitance is attained through the deposition of graphite. In this way neither the electric resistance nor the mechanical strength of the metal foil are impaired.

DETAILED DESCRIPTION OF THE INVENTION

More specifically, the present invention provides a process for producing an impermeable or substantially impermeable electrode suitable for use in an electrolytic capacitor or battery,

which comprises immersing a substrate in a suspension comprising graphite in an organic solvent in a concentration of graphite of 1 to 50 g/l to deposit a layer of graphite on the substrate, removing the substrate with graphite layer thereon from the suspension, drying the substrate with graphite layer thereon at approximately 80 to 150°C, and heat-treating the dried substrate with graphite layer thereon at 200 to 450°C to form an impermeable or substantially impermeable conductive layer of graphite on the substrate.

The invention provides in particular a simple and effective process for producing an electrode for electrolytic capacitors, supercapacitors or batteries with an impermeable conducting layer of graphite, which is deposited from a suspension comprising graphite at a concentration between 1 and 50 g/l in an organic solvent onto a substrate by immersion for a given length of time of, for example, approximately 10 to 60 seconds, wherein, after the deposition, the substrate with the layer comprising graphite is dried at a temperature between 80 and 150 °C for a given length of time of, for example, approximately 1 minute and, after the drying, is heat-treated at a temperature between approximately 200 and 450 °C for a given length of time, for example, approximately 5 to 60 minutes.

The organic solvent which serves as a vehicle for the graphite during the layer formation is eliminated during the drying. In the final heat treatment at increased temperatures a compact impermeable deposition layer of graphite is obtained, which covers the substrate and adheres to it. The selection of the temperature and length of the heat treatment determines the quality of the compactness and of the adhesive strength of the layer.

If the capacitance of such an electrode is measured in a solution of ammonium adipate with a resistance of 15 Ω.cm and a pH value of 6.6, considerable capacitance values between

100 μF and 5000 μF are obtained.

If, for example, an etched cathode foil having a thickness of 50 μm (length of the substrate: 100 cm; width of the substrate: 4 cm), such as is conventionally used in capacitor technology, is compared in the usual manner with a corresponding foil of 30 μm thickness, on which a layer of graphite has been deposited according to the invention, the results in Table 1 are obtained:

Table 1

Foil thickness	Technology	Electric resistance	Capacitance μF/cm^2	Mechanical strength
30 μm	produced according to the invention	30 mΩ	1000	35 N/cm
50 μm	conventionally etched	33 mΩ	440	30 N/cm

It is evident that with the process according to the invention significantly higher capacitance values with considerably lesser foil thickness can be attained, and specifically at lower electric resistance and higher mechanical strength. Electrodes according to the invention are therefore far superior to conventional etched foils. Due to the lower material strength of the electrodes according to the invention it is possible, for example when they are used as a cathode in an electrolytic capacitor, to achieve significantly higher capacitance values at identical volume.

The following Table 2 shows the characteristic capacitance behavior with respect to the frequency of an electrode (approximately 20 cm^2) according to the invention in comparison to a cathode etched in the conventional manner.

Table 2

Frequency (Hz)	Electrode produced according to to the invention μF	Conventionally etched cathode μF
10	80000	4400
20	40000	4000
50	18000	3600
100	9600	3400
1000	1100	2500
10000	50	1010

It is evident that electrodes according to the invention assume considerably higher capacitance values at low frequencies below approximately 300 Hz. Capacitors equipped with electrodes according to the invention are therefore preferably suitable for energy storage.

The substrate for the electrode according to the invention on which the graphite is deposited, is preferably a metal foil, for example comprising aluminum, or an insulating foil comprising a synthetic material. When using a metal foil, the layer of graphite behaves like a short circuit and prevents any change in the ohmic resistance of the substrate. If the substrate is insulating, the graphite layer represents a minimum electric resistance. The metal foil can remain untreated before the deposition of the graphite, however, it is preferably chemically or electrochemically pickled and/or treated with mechanical surface treatment, such as brushing, in order to further improve the efficiency of the electrode produced according to the invention.

The layer of graphite can be deposited on one side or on both sides of the substrate.

The organic solvent for the graphite suspension is, for example, an alcohol, a mixture of alcohols or a solvent having a carbonyl group.

The heating of the layer of graphite should preferably take place in a controlled atmosphere or in an inert gas atmosphere such as a nitrogen or argon atmosphere.

The substrate for the deposition of the graphite preferably has a thickness between approximately 15 and 55 μm.

The invention is also directed to the use of an electrode produced according to the previously described process. The use takes place, as already mentioned, preferably as a cathode of an electrolytic capacitor for very low frequencies, which has an anode supporting an oxide layer with dielectric properties, for example as an electrode of a supercapacitor operating according to the principle of the Helmotz double layer and a diffusion layer.

The electrode produced according to the invention can also be used as the negative electrode of a battery.

When using the electrode in a graphite battery with a graphite block and a negative case, a metal foil serves as the substrate, both sides of the substrate are covered with a layer of graphite, and a contact between the graphite block and the negative case is established.

When using an electrode according to the invention as the electrode of a lithium battery, the substrate is a metal foil which is placed between the separators and is connected with the negative battery case.

IN THE CLAIMS

1. A process for producing an impermeable or substantially impermeable electrode suitable for use in an electrolytic capacitor or battery, which comprises immersing a substrate in a suspension comprising graphite in an organic solvent in a concentration of graphite of 1 to 50 g/l to deposit a layer of graphite on the substrate, removing the substrate with graphite layer thereon from the suspension, drying the substrate with graphite layer thereon at approximately 80 to 150°C, and heat-treating the dried substrate with graphite layer thereon at 200 to 450°C to form an impermeable or substantially impermeable conductive layer of graphite on the substrate.

2. The process according to claim 1, wherein the substrate is immersed in the suspension for approximately 10 to 60 seconds.

3. The process according to claim 1, wherein the drying is for approximately 1 minute.

4. The process according to claim 1, wherein the heat-treating is for approximately 5 to 60 minutes.

5. The process according to claim 1, wherein the substrate is a metal foil, or an insulating foil comprising a synthetic material.

6. The process according to claim 5, wherein the metal foil is an aluminum foil.

7. The process according to claim 5, wherein the metal foil, before immersion in the suspension, is untreated, chemically treated, electrochemically pickled, or subjected to mechanical surface treatment.

8. The process according to claim 7, wherein the mechanical surface treatment is brushing.

9. The process according to claim 1, wherein a layer of the graphite is deposited on one side of the substrate.

10. The process according to claim 1, wherein a layer of the graphite is deposited on both sides of the substrate.

11. The process according to claim 1, wherein the organic solvent is an alcohol, a mixture of alcohols, or a carbonyl group-containing organic solvent.

12. The process according to claim 1, wherein the heat-treating is conducted in a controlled atmosphere or an inert gas atmosphere.

13. The process according to claim 12, wherein the inert gas atmosphere is a nitrogen or argon atmosphere.

14. The process according to claim 1, wherein the substrate has a thickness of approximately 15 to 55 μm.

15. An impermeable or substantially impermeable electrode suitable for use in an electrolytic capacitor or battery, which comprises a substrate with an impermeable or substantially impermeable conductive layer of graphite on the substrate.

16. An electrolytic capacitor comprising, as a cathode, a substrate with an impermeable or substantially impermeable conductive layer of graphite on the substrate, and an anode which has an oxide layer with dielectric properties.

17. The capacitor according to claim 16, which is a supercapacitor which operates according to a principle of a Helmotz double layer and a diffusion layer.

18. A battery comprising, as a negative electrode, a substrate with an impermeable or substantially impermeable conductive layer of graphite on the substrate.

19. The battery according to claim 18, which is a graphite battery having a graphite block and a negative battery case, and wherein the substrate is a metal foil, both sides of the metal foil are covered with a layer of the graphite, and contact is established between the graphite block and the negative case.

20. The battery according to claim 18, which is a lithium battery having separators and a negative battery case, and wherein the substrate is a metal foil placed between the separators and connected with the negative battery case.

21. Process of manufacturing an impermeable electrode for electrolytic capacitors, supercapacitors and batteries, providing an impermeable conductive graphite layer on a substrate, deposited from a suspension of graphite having a concentration of the graphite in its solvent between 1 and 50 g/1, by immersing the substrate into the suspension for a predetermined time of e.g. approximately 10 to 60 seconds, the deposition of the graphite layer being followed by drying the substrate with the deposited layer at a temperature between 80 and 150°C for a predetermined time of e.g. approximately 1 minute and being followed by thermal treatment at a temperature between 200 and 450°C for a predetermined time of e.g. approximately 5 to 60 minutes.

22. Process according to claim 21, wherein a metal foil, e.g. aluminum foil, or an isolating foil is used as a substrate.

23. Process according to claim 22, wherein the metal foil before the deposition of the graphite layer is kept raw or surfaced pickled, carried out chemically or electro-chemically, and/or mechanically surfaced treated, e.g. by brushing.

24. Process according to claim 21, wherein the graphite layer is deposited on one side or on both sides of the substrate.

25. Process according to claim 21, wherein alcohol, a mixture of alcohols or a solvent comprising substances with carbonylic groups is/are used as organic solvent for the graphite.

26. Process according to claim 21, wherein the thermal treatment of the graphite layer is performed in a controlled atmosphere or an inert gas atmosphere, as nitrogen or argon.

27. Process according to claim 21, wherein a substrate is used having a thickness of about 15 to 55 μm.

28. Use of an electrode as described in claim 21 as a cathode of an electrolytic capacitor having an anode carrying a layer of an oxide with dielectric characteristics deposited on it, or as an electrode of a supercapacitor working according to the principle of Helmotz double layer and diffusion layer.

29. Use of an electrode as described in claim 21, as a negative electrode in an electric battery.

30. Use according to claim 29, as an electrode in a graphite battery having a graphite block and a negative casing, wherein a metal foil is used as substrate, wherein both faces of the substrate are coated by a graphite layer and wherein electrical contact is insured between the graphite block and the negative casing.

31. Use according to claim 29, as an electrode in a lithium battery, wherein a metal foil is used as a substrate, which is inserted between the separators and connected to the negative battery casing.

ABSTRACT OF THE DISCLOSURE

A process for producing an impermeable electrode for electrolytic capacitors, supercapacitors or batteries, with an impermeable conductive layer of graphite, which is deposited from a suspension comprising graphite at a concentration between 1 and 50 g/l in an organic solvent on a substrate by immersion for a given length of time of, for example, approximately 10 to 60 seconds and wherein, after the deposition, the substrate with the layer of graphite is dried at a temperature between approximately 80 and 150 °C for a given length of time of, for example, approximately 1 minute and, after the drying, is heat-treated at a temperature between approximately 200 and 450 °C for a given length of time of, for example, approximately 5 to 60 minutes.

DECLARATION AND POWER OF ATTORNEY FOR U.S. PATENT APPLICATION

(X) Original ( ) Supplemental ( ) Substitute ( ) PCT ( ) Design

As a below named inventor, I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that I verily believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Title: PROCESS FOR PRODUCING AN ELECTRODE AND USE OF THE ELECTRODE

of which is described and claimed in:
( ) the attached specification, or
(x) the specification in the application Serial No. _____ filed July 20, 1999 ;
and with amendments through _____ (if applicable), which is based on
( ) the specification in International Application No. PCT/_____, filed _____, and as amended on _____ (if applicable).

I hereby state that I have reviewed and understand the content of the above-identified specification, including the claims, as amended by any amendment(s) referred to above.

I acknowledge my duty to disclose to the Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim priority benefits under Title 35, United States Code, §119 (and §172 if this application is for a Design) of any application(s) for patent or inventor's certificate listed below and have also identified below any application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

COUNTRY	APPLICATION NO.	DATE OF FILING	PRIORITY CLAIMED
Germany	198 32 355.7	July 20, 1998	Yes

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION SERIAL NO.	U.S. FILING DATE	STATUS: PATENTED, PENDING, ABANDONED

And I hereby appoint Michael R. Davis, Reg. No. 25,134; Matthew M. Jacob, Reg. No. 25,154; Jeffrey Nolton, Reg. No. 25,408; Warren M. Cheek, Jr., Reg. No. 33,367; Nils E. Pedersen, Reg. No. 33,145; and, Charles R. Watts, Reg. No. 33,142, who together constitute the firm of WENDEROTH, LIND & PONACK, L.L.P., jointly and severally, attorneys to prosecute this application and to transact all business in the U.S. Patent and Trademark Office connected therewith.

I hereby authorize the U.S. attorneys named herein to accept and follow instructions from KEIL & SCHAAFHAUSEN as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. attorneys and myself. In the event of a change in the persons from whom instructions may be taken, the U.S. attorneys named herein will be so notified by me.

Send Correspondence to

WENDEROTH, LIND & PONACK, L.L.P.
2033 K Street, N.W., Suite 800
Washington, D.C. 20006

Direct Telephone Calls to:

WENDEROTH, LIND & PONACK, L.L.P.
Area Code (202) 721-8200

Direct Facsimile Messages to:
Area Code (202) 721-8250

Full Name of First Inventor	FAMILY NAME CHIAVAROTTI	FIRST GIVEN NAME Giovanni	SECOND GIVEN NAME Pietro
Residence & Citizenship	CITY Milano	STATE OR COUNTRY Italy	COUNTRY OF CITIZENSHIP Italy
Post Office Address	ADDRESS Via Forze Armate, 260/5, I-20152 Milano, Italy	CITY	STATE OR COUNTRY ZIP CODE
Full Name of Second Inventor	FAMILY NAME CONSTANTI	FIRST GIVEN NAME Jean	SECOND GIVEN NAME
Residence & Citizenship	CITY Poissy	STATE OR COUNTRY France	COUNTRY OF CITIZENSHIP France
Post Office Address	ADDRESS 5, Rue de Strasbourg, FR-78300 Poissy, France	CITY	STATE OR COUNTRY ZIP CODE
Full Name of Third Inventor	FAMILY NAME VONO	FIRST GIVEN NAME Giuseppe	SECOND GIVEN NAME
Residence & Citizenship	CITY Milano	STATE OR COUNTRY Italy	COUNTRY OF CITIZENSHIP Italy
Post Office Address	ADDRESS Via Dei Pini, 2/M, 20090 Pieve Emanuele, Milano, Italy	CITY	STATE OR COUNTRY ZIP CODE
Full Name of Fourth Inventor	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
Residence & Citizenship	CITY	STATE OR COUNTRY	COUNTRY OF CITIZENSHIP
Post Office Address	ADDRESS	CITY	STATE OR COUNTRY ZIP CODE

Full Name of Fifth Inventor	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME	
Residence & Citizenship	CITY	STATE OR COUNTRY	COUNTRY OF CITIZENSHIP	
Post Office Address	ADDRESS	CITY	STATE OR COUNTRY	ZIP CODE
Full Name of Sixth Inventor	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME	
Residence & Citizenship	CITY	STATE OR COUNTRY	COUNTRY OF CITIZENSHIP	
Post Office Address	ADDRESS	CITY	STATE OR COUNTRY	ZIP CODE
Full Name of Seventh Inventor	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME	
Residence & Citizenship	CITY	STATE OR COUNTRY	COUNTRY OF CITIZENSHIP	
Post Office Address	ADDRESS	CITY	STATE OR COUNTRY	ZIP CODE

I further declare that all statements made herein of my own knowledge are true, and that all statements on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

1st Inventor Gianpietro Chiavarotti Date AUGUST 10TH - 1999

2nd Inventor Giovanni Pietro CHIAVAROTTI Jean Constanti Date August 10th 1999

3rd Inventor Jean CONSTANTI Giuseppe Vono Date AUGUST 10TH - 1999

Giuseppe VONO

4th Inventor _____ Date _____

5th Inventor _____ Date _____

6th Inventor _____ Date _____

7th Inventor _____ Date _____

The above application may be more particularly identified as follows:

U.S. Application Serial No. 09/357,300 Filing Date July 20, 1999

Applicant Reference Number B 4 P 16 US Atty Docket No. 159/B4P16US

Title of Invention PROCESS FOR PRODUCING AN ELECTRODE AND USE OF THE ELECTRODE